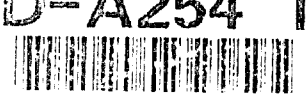

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NAVSWC MP 91-678

PORTABLE FTIR SPECTROMETER OPERATING MANUAL

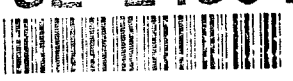
BY JEFFRY J. FEDDERLY

RESEARCH AND TECHNOLOGY DEPARTMENT

16 DECEMBER 1991

NAVAL SURFACE WARFARE CENTER

Dahlgren, Virginia 22448-5000 • Silver Spring, Maryland 20903-5000



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FOREWORD

Fourier transform infrared (FTIR) spectroscopy has proved to be a valuable tool in the analysis of materials. While originally developed as a laboratory research instrument, more recently FTIR has found applications in factory environments, such as in steel mills. Work done over the past several years at the Naval Surface Warfare Center (NAVSWC) has demonstrated the usefulness of this instrument for quality control purposes. Because the instrument might only be used on an occasional basis at various locations during different stages of a program, it is desirable that the instrument be made easily portable and usable by personnel from different facilities with a minimum of training. This manual is intended to accompany the packaged instrument. It contains instructions for unpacking, setup, operation, and repacking for shipment.

The appendix dealing with mirror alignment is reproduced from information supplied by Spectra-Tech, Inc., the manufacturer of the horizontal attenuated total reflectance (ATR) attachment to the FTIR.

While the development of the FTIR instrument for this purpose was carried out over the past several years, the preparation of this manual was done during Fiscal Year 1992 under support from the Naval Sea Systems Command.

Approved by:

Carl E. Mueller

CARL E. MUELLER, Head
Materials Division

ABSTRACT

This manual contains detailed instructions for the operation of a Fourier Transform Infrared (FTIR) Spectrometer. The instructions cover the installation of the spectrometer and its associated computer after shipment, the operation of the instrument, and repackaging for shipment. Some hints on troubleshooting are also provided.

CONTENTS

INTRODUCTION

This manual provides information for the installation and operation of the Mattson Instruments Fourier Transform Infrared (FTIR) Spectrometer. The use of the spectrometer with an attenuated total reflectance (ATR) attachment is used to determine the isocyanate (NCO) index of polymer materials. Please read through the entire manual before beginning the installation or operation of this instrument. Then, as the instrument is being installed and operated, read each section thoroughly again. This manual is sufficiently detailed for a smooth setup and operation of the spectrometer. This manual is written under the assumption that the person installing the computer software has some basic understanding of the disk operating system (DOS) and is capable of making and changing directories, editing the autoexec.bat file, etc. The verbal descriptions are adequate for an operator to use without FTIR experience.

The spectrometer is a Mattson Galaxy 4021 with a Mercury-Cadmium-Telluride (MCT) detector and 1 cm^{-1} resolution capability. The spectrometer can be controlled by any IBM-AT compatible "286" or "386" computer via a standard RS-232 interface. With the use of a custom-made shipping case, the instrument is quite portable. It is not possible, however, to ship the spectrometer completely "ready to go." It is necessary to package delicate and hygroscopic components separately. The ATR attachment comes installed in the sample compartment of the spectrometer, but it is necessary to temporarily remove it during the set-up procedure. This manual discusses the unpacking and installation of these components, along with some optical realignment that may be necessary. The parts list for shipping is given in Appendix A.

Computer software is provided that allows for both the general operation of the instrument as well as for the specific quality control (QC) analysis. The manual describes the loading and operation of this software. The general operational and analysis software is Mattson Instrument's FIRST™ software package. This package has many capabilities that are not discussed in this manual. Only the portions necessary for the basic operation of the instrument will be discussed. The specific data acquisition and analysis tasks needed for testing a specific material are performed by macro programs written by Naval Surface Warfare Center (NAVSWC). These macros allow for automated data collection and analysis and are independent of the FIRST™ software.

A considerable amount of care must be made when loading the samples onto the zinc selenide (ZnSe) ATR crystal. If the sample is not loaded properly or too much force is used, the crystal will crack. Replacing damaged crystals is an expensive proposition. Take care in reading the section on loading the ATR samples.

INSTALLATION

FTIR SPECTROMETER

General Care and Concerns

Although the instrument is designed to operate in a fairly rugged environment, some special care and precautions should be taken. This is especially true while installing the instrument when more of the optics are exposed. A potassium bromide (KBr) beamsplitter and a KBr window need to be installed. Be careful not to touch or scratch the faces of these parts. As these parts are installed, covers that normally protect other optical devices and mirrors are removed. Be especially careful not to touch or scratch any of these parts as well. The mirrors on the instrument and on the ATR device are highly polished and unprotected. **Please do not attempt to clean any of the mirrors.** Dust on the mirrors affects instrument performance very little. While the instrument is being set up, the operator is also more exposed to the laser beam. This is not an extremely powerful laser and it generally will not be harmful, but exercise caution when installing and operating the spectrometer. Do not look directly into the laser or along the path of the beam. The red dots of light that you see on some of the optics is from the laser.

The KBr beamsplitter and the KBr window are hygroscopic and need to be kept fairly dry. The interferometer chamber where these parts are located is desiccated to a certain degree; **but it is also best, once the instrument is installed, to leave the instrument turned on at all times.** The heat from the infrared source also serves to keep these items dry.

Unloading the Spectrometer

Before unloading the fiberglass shipping case containing the spectrometer, loosen the pressure relief valve located on one of the sides. Instructions are given on the case adjacent to the valve. Open the container by lifting up the flaps on the 12 metal latches and turning them 1/2 turn counterclockwise. Lift off the cover noting the orientation of the foam cutout in relation to the raised surface on the spectrometer. There are two handhold cutouts on the bottom of both the left- and right-hand sides of the spectrometer. Have two people reach between the spectrometer and the foam insulation to locate these handholds and lift the spectrometer out. Note the position that the spectrometer was placed in the container and repack in the same manner. Set the spectrometer on a solid level surface for setup and operation.

In the event that the spectrometer is to be used in an area where there is no bench space, the shipping case doubles as a portable bench. Remove the four telescoping legs from the case and reattach the top of the case to its base. The legs can be attached to the bottom of the case by inserting them through the holes in the runners and securing with the pins. There is a bit of excess play in the legs, but that can be remedied with some shims to fill the gaps. The spectrometer is at a pretty good working height with the legs fully contracted. The telescoping legs can then be used to compensate for an uneven floor or ground.

Make certain that all of the packing containers and special packaging items are retained for use in repacking the instrument. Place any of the bags containing desiccant in a desiccator or another tightly sealing container.

Install Cables

Position the spectrometer so that you have access to the lower rear panel of the instrument. Locate the power cable and RS-232 cable from the parts box. Plug the power and RS-232 cables into the instrument. The RS-232 cable is labeled as to which end to attach to the instrument. It is not necessary to plug the cables into the wall outlet and computer at this time; it is just easier to install the cables at the rear of the instrument before you proceed with the rest of the installation. Reposition the instrument to the desired operating location.

Remove the Spectrometer Cover

Removing the main cover of the spectrometer gives you access to the interferometer housing and many spectrometer optical elements.

1. Two 1/4-turn fasteners are located on the spectrometer's rear panel in each upper corner. Turn these fasteners 1/4 turn in a counter-clockwise direction as you face the rear of the spectrometer or in a clockwise direction as you face the front of the spectrometer.

2. Release the two slide clamps located on the outsides of the front lip of the spectrometer baseplate. Slightly compress the front of the cover while sliding the clamps towards the center of the spectrometer.

3. Slide the cover forward 2 to 4 inches. The forward motion of the cover may be hindered by the ATR unit that is installed in the sample compartment. You may need to lift up slightly on the instrument cover as you slide it forward or else open the sample compartment cover momentarily to keep the cover from hanging up on the ATR unit.

4. Lift the spectrometer cover straight up and away from the spectrometer. Reverse these steps for replacing the cover.

Unlock the Interferometer Moving Mirror

The center portion of the spectrometer has another cover which houses the interferometer and infrared source. On the top of this cover is a ring handle which is used to lock and unlock the moving mirror. The mirror is locked for transit and needs

to be unlocked for use. Pull the ring from the lowered locked position to the upper unlocked position as shown in the diagram on the cover.

Install the Interferometer Cover Window

Remove the interferometer chamber cover by removing the two knurled nuts on the top of the cover. Remove the ribbon cables that are attached to the sides of the cover, then lift the cover straight out. To remove the cover, you can use the cable holders on the right-hand side to grab on to. There is really nothing to hold on to on the left-hand side. Just put pressure on the lower portion of the cover and jiggle the cover as you lift it out. On the front of the cover, there is an open window surrounded by a ring held in place by three nuts. Remove the nuts and the outer ring. Leave the foam ring in place. Now find the package marked **Interferometer Window** in the parts box and carefully remove the 50-mm KBr window from the metal box. Being careful not to touch the faces of the disk, place it over the foam ring on the cover. You may rest the disk on the two bottom bolts that extend from the cover. Making sure that the disk does not fall over, replace the outer ring and gently tighten the three nuts to compress the foam slightly to make a seal. You can use the supplied needlenose pliers or a small wrench to tighten the nuts if finger tightening is insufficient. Do not replace the cover yet.

Install the Interferometer Desiccator Units

Remove the tape on the interferometer cover where it reads **Interferometer Desiccators**. Locate the two cylindrical desiccator units from the parts box. If the indicators at the tops of the units are not light blue, the desiccant in the tubes can be dried according to the set of instructions located on the top of the cover. To install the tubes, unscrew the bottom nut and remove the nut and bottom gasket from each unit. Drop the units into the holes at the top of the cover and secure with the gaskets and nuts from the inside of the cover.

Install the KBr Beamsplitter

While the interferometer cover is off, the KBr beamsplitter needs to be installed. Find the package marked **Beamsplitter** in the parts box and carefully remove the part from the packaging. Carefully remove the plastic plates that are protecting the window faces of the beamsplitter. Make certain that the window portions of the beamsplitter are not scratched or touched in any way. Remove the tape that holds the two screws in place. To install the part, place the three pins on the bottom of the beamsplitter into the matching holes in the center of the interferometer base. The writing on the top of the part needs to face the front left corner of the spectrometer. Now tighten down the two screws with the 5/32-inch allen wrench that is provided to secure the beamsplitter. Replace the interferometer cover, making sure that none of the cables on any of the sides interfere with the cover going down completely. Replace the two knurled nuts and snug down slightly. Place the ribbon cables back into their holders.

Cool the Detector

In order for the spectrometer to function, the MCT detector must be cooled with liquid nitrogen. You will need to have on hand a dewar filled with about half a liter, or slightly more. Open the black cover located in the front left-hand portion of the instrument. Find the plastic funnel in the parts box and insert it into the hole at the top of green detector. Fill the funnel and let it drain. Repeat this once or twice then wait for the plume to die down. Repeat the above procedure. If the nitrogen starts to spill over the top of the detector, remove the funnel from it and let the plume die down. It is best not to have too much nitrogen spilling over the front of the detector where the window is located. Now, top off the detector by slowly adding nitrogen until the detector is filled. The very top portion of the detector will not stay filled for more than a few seconds. This method takes a few minutes to complete, but it is the best way if you want the detector to stay cooled all day.

If the spectrometer only needs to be used for a short while, it will be quicker to simply fill the detector with about four funnels full until it starts to overflow. Remove the funnel. For best results, wait for about 15 minutes after filling before using the spectrometer.

Remove Horizontal ATR Unit

The spectrometer was shipped with the ATR unit installed into the sample compartment. For the alignment procedure that will be performed in the next section, remove the ATR unit. Remove the single screw that holds down the bottom base plate with the 9/64-inch allen wrench. (*It is not necessary to remove the two screws that hold the main ATR unit to the ATR base plate.*) Lift the unit straight out and place it in a safe place.

Align Interferometer Mirror

Every time the spectrometer is transported and the beamsplitter removed and replaced, a slight realignment of the optics will be necessary. Generally, the only alignment needed is an adjustment of the interferometer's stationary mirror. For this adjustment, the instrument needs to be turned on. Before turning on the instrument, make sure that the moving mirror has been unlocked. Plug the power cord into a standard 115-volt wall outlet and turn the instrument on. The on/off switch is a vertically positioned rocker switch located on the left-hand side of the rear panel just to the left of the power cable outlet as you face the front of the instrument. The RS-232 cable need not be connected to the computer at this time.

Once the instrument is turned on, check two things before proceeding with the alignment. Look into the right-hand window on the top interferometer cover. You should see an orange glow emitted from the round face of the infrared source. If there is no orange glow, the source is probably burned out and will need to be replaced. Next, look into the left-hand window. You should see several dots of red laser light being imaged on the beamsplitter. If there are no red dots, the laser is probably burned out and will need to be replaced. Also, if the alignment is not too bad, the moving mirror will be scanning back and forth, and you will be able to see this in the left-hand window.

If the mirror is moving, after a self diagnostic period, the display should read "Ready to collect spectrum" at the top. The "scan" light should be flashing yellow; the green "ready" light should be lit as well as the yellow "reference" light. To perform the alignment, press the **Tune** button on the contro[1] panel of the spectrometer. The display on the left shows you the current detector voltage, and the display on the right shows the maximum voltage seen since the tune session began. The detector voltage is your guide to the quality of the signal strength and optical alignment. Unscrew the two red caps on the top of the interferometer cover. This gives access to two micrometers that are used to align the stationary mirror. Insert the 5/32-inch allen wrench into the slot on top of the left micrometer and slowly adjust the micrometer until the voltage, shown in the Peak display, is maximized. Now do this for the right micrometer. Repeat this procedure once or twice to optimize the voltage. One should be able to obtain voltages of 5 volts or better. Replace the red caps after the alignment has been completed.

If the mirror does not begin to scan after the spectrometer is turned on, the alignment is probably too poor for the detector to receive any signal. Access the micrometer adjustments as described in the previous paragraph. Randomly adjust the two micrometers until the detector receives enough signal and the mirror starts moving. Once the mirror starts scanning, optimize the alignment following the procedure described in the previous paragraph. If after repeated attempts you cannot get the mirror to scan, either the detector is defective or something else in the optical system is grossly out of alignment.

If, after several attempts to adjust the micrometers, it appears that they are more out of alignment than when you started, the micrometers can be returned to the settings that they were shipped with. The left micrometer had a value of 1.51, and the right micrometer had a setting of 1.52. To read the values on the micrometer barrels, the interferometer cover must be removed again. The beamsplitter and interferometer mirrors are now exposed. Be very careful and do not leave the cover off for extended periods of time.

Replace Spectrometer Cover

Reinstall the spectrometer cover by reversing the instructions from the **Remove Spectrometer Cover** section. Be careful to tuck in any loose cables extending from the base. Once the cover is in its proper position, remember to compress the top of the instrument slightly in order to more easily lock the slide clamps.

ATR Mirror Alignment

With the instrument display still in the tune mode, note the open compartment voltage. Open the sample compartment cover and reinstall the ATR unit into the sample compartment and secure with the machine screw. Locate the box in the parts box labeled **ATR Plate** and carefully remove the ZnSe plate. Insert the five pins of the plate into the holes at the top of the ATR unit. Note the voltage on the tune display. With a new crystal and optimum alignment of the ATR mirrors, a voltage of about 50 percent of the open compartment voltage can be obtained. If the reading is less than 25 percent of the original voltage, the mirrors of the ATR unit should be realigned. If realignment is necessary, follow the alignment procedure given in Appendix B, which has been reproduced from the Spectra-Tech CONTACT

SAMPLER™ operators manual. Spectra-Tech cites lower acceptable voltage percentages, but the higher values are readily obtainable.

Take the spectrometer out of the tune mode by pressing the **Stop/Reset** button on the spectrometer control panel.

ATR Gripper Installation

Close the sample compartment cover and locate the package labeled **Gripper** from the parts box. Remove the tape from the two screws on the sides of the mounting block. With the 5/32-inch allen wrench, insert the screws into the threaded holes on the top of the ATR unit and snug firmly. The swinging pressure plate with the rubber backing should extend over the ATR crystal.

Note: With the Gripper device installed, the sample compartment cover cannot be opened fully. Therefore, the detector cannot be filled with liquid nitrogen without first removing the Gripper device.

COMPUTER

Requirements

The computer must be 100 percent IBM PC-AT compatible, operating with DOS version 3.3 or higher. A "386" computer is preferable, but satisfactory operation is obtained with a "286" machine. The computer should be equipped with 1 Megabyte (MB) of Random Access Memory (RAM) and a math co-processor. Installation of the software will require slightly more than 2 MB of disk space. Every spectrum that is saved will require about 15K of disk space. Make sure that you have enough free space on the hard drive for loading the software and storing a few spectra. For the RS-232 connection to the spectrometer, one free 9-pin serial port is required. A Microsoft bus mouse is required to operate the Mattson FIRST™ software.

Mouse

If a Microsoft bus mouse is not already installed in the computer, install the provided mouse and mouse driver software according to the manufacturer's instructions.

Cables

Find the end of the RS-232 cable marked "computer" and install it into the 9-pin serial port. If the other end has not already been connected to the spectrometer, do it now.

Install the Software

After booting up the computer, insert the disk labeled Enhanced FIRST™ into the 5.25-inch "A" drive. At the DOS prompt, type **a:install**. The only response from

you is to answer the question about the spectrometer type. Respond with **40xx**. Next, insert the disk labeled FIRST™ Macros into the A drive. Again, type **a:install**. The Mattson Instruments software has now been installed. To access the software from any directory, you will have to modify the path command in the autoexec.bat file to include, **c:\first\bin;** in the list of directories to search. The installation program does not do this for you.

Insert the disk labeled "Macro Programs" into the A drive. This disk contains the NAVSWC written macros and other pertinent files for use in collecting and analyzing the data. These macro programs need to be accessed from the same directory in which they are stored. Copy the contents of this disk into the directory where you plan on storing the spectra. You may wish to create a new directory that will contain just stored spectra and the macro programs. Change directories to the desired directory and type **copy a:\ratio*.***. This will copy the macro programs and an example spectrum file into the desired directory.

With the same disk in drive A, move to the \FIRST\CFG directory. Type **copy a:\method*.***. All of the software has now been installed and the system should be ready for operation.

OPERATION

This section describes the series of steps necessary for the operation of the instrument, once it has been installed.

PREPARE INSTRUMENT

Fill the detector with liquid nitrogen, close the sample compartment cover, and install the "Gripper" device according to the installation instructions. Verify that the spectrometer is scanning (scan light flashing on and off) and that everything appears satisfactory for operation. After 15 minutes, run the **tune** routine to check that approximately the same voltage is obtained as was obtained when installing the instrument. Be sure to press the **stop/reset** button in order to exit the tune mode before running the macro program.

SAMPLE PREPARATION

Cut the samples to be tested from a 2-inch x 12-inch x 0.080 inch molded test sample. Use a razor blade or similar instrument. Allow the sample to cure for at least 2 hours prior to testing. The sample should have a length of 2.75 inches by about 0.5 to 0.6 inch. The cast faces of the sample to be tested should be mirror-like and free of physical defects. Wipe the sample with a dry laboratory wipe to remove any mold release agent, fingerprints, or other contaminants.

ATR SAMPLE LOADING

The following procedure should be followed any time the macro program calls for a sample scan or any time that you are acquiring a sample spectrum with the Mattson FIRST™ software. In addition to supplying a good ATR crystal, a previously cracked one is also provided. This will: (1) show you what happens when a sample is loaded improperly and (2) provide you with a crystal to practice getting a feel for the loading procedure.

Note: You will not be able to obtain a reasonable spectrum with the cracked crystal.

The ATR Gripper device should be properly adjusted for a 0.070- to 0.080-inch thick sample. The distance between the bottom of the black plate and the top of the silver pressure plate (the length of the springs) should be 0.70 inch. Check to see if the device is set properly and that it is the same on both ends. The micrometer at the top of the device should be set to 3.75. These settings should allow for a 70-mil sheet to be compressed slightly.

Center a sample on the ZnSe ATR crystal. To lower the pressure pad onto the sample in order that the sample makes good contact with the crystal, the black handle that is facing you must be pushed up towards the micrometer handle. If the plate is lowered straight down onto the sample, the tendency, as the sample is compressed, is for the front of the plate to pitch downward. When this occurs, the sample is compressed unevenly and poor contact is made with the crystal.

As the plate is lowered, tilt the plate slightly towards you such that the front of the pressure plate extends just slightly beyond the front of the ATR crystal plate as contact is made. Hold that position with one hand as you finish lifting the handle with the other. Lift the handle until it snaps into place. As you are squeezing the sample, a moderate amount of resistance will be felt. The crystal can take a fair amount of pressure, but excessive pressure will cause it to crack. The greatest stress on the crystal will be when the plate is being rolled over the sample upon loading. Therefore, **if you feel excessive resistance as you are loading the sample, stop and back off on the micrometer setting (turn counter-clockwise), before continuing.** After the handle has been fully lifted, check to see that the sample has been loaded evenly. To do this, place the small level provided on top of the pressure plate and verify that it is reasonably level in relation to the ATR crystal plate. If it isn't, release the pressure plate and try again. After the plate has been lowered and leveled, additional pressure can be applied by tightening the micrometer (clockwise) until it is finger tight using the thumb and forefinger.

After the FTIR scan is complete, gently release the handle by pulling down on it. **Loosen the micrometer to its initial setting.** The original setting for the micrometer was given as a guideline; thicker samples will require a looser (larger number) setting and thinner samples will require a tighter setting. This is a procedure that requires a fair amount of practice. Quite a bit of pressure is needed in order to acquire a sufficiently good spectrum for the QC analysis, yet too much pressure being exerted while the plate is rolling over the sample upon loading can cause the crystal to crack. Practice with lower loading pressures until you get a feel for the procedure.

ACQUISITION AND ANALYSIS

A macro program has been written which allows for automated data acquisition and quantitative analysis. The program automatically sets up the proper parameters for data acquisition and initializes the spectrometer. It also prompts the operator for the collection of the background and sample scans and then performs the data analysis.

To invoke the macro, move to the directory where the macro programs were stored (see the section on software installation). At the DOS prompt, type **firstm**. This brings you into a FIRST™ macros environment from which either macro programs or individual commands can be run. Before the macro is run, make sure that the detector is cooled and that the ATR unit is installed and ready to operate.

To start the program, type **run ratio-a** at the **firstm** prompt. The program will initialize the spectrometer and then prompt the operator to prepare for a background scan. Before running the background, make sure that the crystal is clean and that nothing is resting on top of it. Press any key to begin the background scan. After the background scan is complete, the operator will be prompted for the sample scan.

Load a sample according to the procedure given earlier. Press any key to start the scan. Once the scan is completed, you may remove the sample. In a few seconds, the spectrum will be created, the peak heights of the two specific peaks will be displayed, and the ratio of these heights will be calculated. The first two values given are the heights of the peaks. This is followed by the peak-height ratio. Record the value of the first (largest) peak height as well as the peak ratio. If the height of the first peak is less than 0.3, the value of the peak ratio is often unreliable. Practice loading the sample and try to maximize the peak heights. The program will ask you whether or not you wish to exit the program. Press y if you wish to exit. Press any key other than y to continue. After four sample scans have been run, the program will prompt you for another background scan and the cycle starts over. When you do press y to exit the program, you will return to the **firstm** prompt. Type **quit** to return to DOS.

If, while running the program, you accidentally made a sample scan when it asked for a background scan, useless results will be generated. After the first real sample scan, exit the program. Type **run ratio-a** again and continue. The program does not store each sample run as a unique file. The data from each run is stored in a file called **tmp1**, and this file is continually being overwritten. You can always look at the last spectrum collected by exiting the macro, calling up the FIRST™ software and choosing **tmp1** as the sample.

ANALYZE STORED DATA

If one wishes to analyze data that was previously collected and stored from the acquisition software (Appendix C), another macro can be used. As before, type **firstm** at the DOS prompt. Type **run ratio-b** at the prompt. Remember that the **ratio-b** macro file must be stored in the same directory as the stored spectra. The program will request a filename. After entering the filename (the .abs extension is not needed), the heights of the two peaks and the peak ratio will be given. If you wish to exit the program, type y. If you wish to continue, press any key other than y.

The file of one NAVSWC-generated spectrum is supplied and should already be stored in the directory that includes the macro programs. The name of this file is **example.abs**. The specimen used to create that spectrum is also supplied. Create new spectra with this sample and compare them to the NAVSWC file.

CALCULATE NCO INDEX

The data for calculating the NCO indices of samples is generated from the macro programs described above. The peak ratio for each sample will be compared to the calibration curve, and the NCO index will be interpolated. The procedure for generating a calibration curve is described in Appendix D. Some guidelines in analyzing the data and determining the peak ratios will follow.

Not all of the spectra taken will be as good as others and, therefore, some of the peak ratios for some scans will not be as good. The best spectra are taken with samples that have very smooth surfaces and that have been evenly loaded onto the crystal. The absolute value of the peak heights of these samples is high. Samples that have a rougher surface, or ones that were loaded onto the crystal poorly, have smaller peaks and generally give poorer results. As a general rule, the first and largest of the two peaks reported should have a value of 0.3 or greater. If the values are lower than this for a sample, try adjusting the way the sample is loaded with the

pressure device. Do not exert excessive force such that the crystal cracks. If it is impossible to obtain a peak height of greater than 0.3 for a given sample, you may have to record and average the values anyway, with the realization that the results may not be as accurate as desired. If you are generally getting values significantly greater than 0.3 for a sample, it is best to throw out results where the peak height is less than that. Even if a value is above 0.3, but is significantly less than the others for that sample, it is a sign that the sample was loaded poorly and should be disregarded.

Average the peak ratio values from at least 10 runs to obtain a sufficiently accurate result for each sample. Calculate the standard deviation (σ) of the peak ratio values. Values that are more than $\pm 2\sigma$ from the mean may be omitted and the standard deviation recalculated. Values for the final standard deviation should be less than 2 percent of the mean, e.g., a σ of less than 0.008 for an average peak ratio of 0.4. If there is more scatter in the data than this, the results are not reliable and more FTIR runs should be made. Once a satisfactory set of peak ratios is obtained, the average value is compared to the standard curve, and the NCO index is interpolated off of the plot.

REPACKING FOR SHIPMENT

This section describes the series of steps necessary for repacking the instrument for shipment.

REMOVING PARTS FROM SPECTROMETER

Remove the ATR crystal plate from the ATR unit. Tape a protective strip of a folded laboratory wipe over the crystal and place in the plastic zip-lock bag. Place the bag, along with plenty of packing material, in the ATR crystal box. Bend the wire ends over to secure the box. Tape another strip over the holes at the top of the ATR unit where the crystal used to be.

Remove the main spectrometer cover and the inner interferometer cover as per the installation. Carefully remove the KBr beamsplitter. Retape the plastic shields over the beamsplitter's windows and secure the screws with tape. Tape the small packet of silica gel to the unit and place the beamsplitter in a plastic zip-lock bag. Place the zip-lock bag with the blue desiccant into the bottom of the plastic container. Set the bag containing the beamsplitter, along with additional packing material, over the desiccant bag and seal the plastic container tight.

Remove the KBr window from the interferometer cover, wrap in the lens tissue, and place in the metal container with the small desiccant package. Seal the container with tape. Replace the interferometer cover and secure it with the two nuts.

LOCK INTERFEROMETER MOVING MIRROR

Lock the interferometer moving mirror from the top of the interferometer cover as shown in the diagram on the cover.

REPLACE COVER AND PACK PARTS

Replace the main instrument cover and secure with the 1/4 turn fasteners at the rear and the slide clamps at the front. Compress the cover slightly in order to engage the slide clamps.

Repack the parts box with all the parts, accessories, tools, and software. Fill the box with additional packing material and secure.

LOAD SPECTROMETER INTO CASE

Make certain that the MCT detector is empty of liquid nitrogen before packing the instrument into the shipping case.

With two people carrying the spectrometer by the handhold cutouts, place the instrument back into the shipping container in the same orientation that it was shipped with. As you are placing the instrument into the container, make sure that the sample compartment cover remains closed and that it gets tucked inside of the foam insulation. Place the top of the shipping case over the instrument. The cutout in the foam should match the raised panel of the instrument. Also, there are matching serial numbers embossed on one side of both the top and bottom halves of the case. These numbers should be aligned if the top is put on in the correct orientation. Secure the two halves together with the 12 latches by engaging the hooks and turning 1/2 turn clockwise. Fold down the flaps and close the relief valve. The instrument is now secured for shipment.

TROUBLESHOOTING

This section will attempt to give a few tips on troubleshooting. Problems that are not readily diagnosed and solved should be directed to Jeff Fedderly, NAVSWC, at (301) 394-2152/1444. Assistance can also be obtained from the Service Department at Mattson Instruments in Madison, Wisconsin. Anyone in the department can be of assistance; however, Mr. Mike Heller is the primary service engineer with whom NAVSWC has dealt. He is aware of the service history on this instrument. The number for Mattson is (608) 831-5515.

In general, most problems with the instrument will be discovered when you initialize the spectrometer. Make sure that the detector has been cooled prior to attempting operation. Most often, there is a bug in the communications between the instrument and the computer. This leads to repeated "The spectrometer is busy" messages on the screen. This may be because the instrument was left in the Tune mode and not reset for regular operation. Even after pressing Stop/Reset, the messages may occur until you receive a "Initialization Failure" message. Press any key to reactivate the screen and try again after resetting the spectrometer from the instruments keypad. Sometimes this will not work and the instrument needs to be turned off for a few seconds and then turned on. Keep track of the messages in the LCD display. After a self-test period, the display should indicate that the instrument is ready to collect spectra and initialization from the computer is possible.

One message that you may encounter is "No signal detected." Check that the detector is cooled and that nothing is blocking the light path. If, after repeated efforts to reset the instrument and/or turning it on and off, the instrument still doesn't respond, remove the main instrument cover and check to see that the source is glowing. If the source is glowing, check to see that the laser is functioning and that the moving mirror is scanning. If either the source or laser is out they will probably need to be replaced. If they are functioning, but the mirror is not scanning, the optics may be way out of alignment. Try adjusting the mirror micrometers according the instructions on page 6. If this does not work, it is possible that either something was knocked out of alignment during shipping or that the detector is not functioning properly. Give either NAVSWC or Mattson a call. A spare source element is provided in the event that the source should burn out.

If a "Laser Fault" message appears, or if the red laser light on the LCD display remains lit, check to see if the laser is operating properly.

Once the spectrometer is installed and functioning properly, very little effort should have to go into maintaining the instrument and very little should go wrong.

APPENDIX A

PARTS LIST FOR SHIPPING

Qty	Description
1	Mattson Galaxy 4021 FTIR Spectrometer NAVSWC P/A 60921067564
1	KBr Beamsplitter
2	KBr Window, 50 mm
2	Interferometer Desiccator Unit
1	Spare FTIR Source Element
1	Spectra-Tech ATR Contact Sampler NAVSWC P/A MINOR103050 (installed in spectrometer)
1	Gripper, Pressure Device for ATR
2	ZnSe ATR Crystal
1	RS-232 Spectrometer/PC Cable
1	RS-232 Spectrometer/Plotter Cable
1	Power Cord
1	FTIR Software Package (3 discs)
1	Microsoft Bus Mouse and Software
1	Test Specimen
1	Funnel
1	Tool Kit, including: 2 long arm allen wrenches, allen wrench set, 2 screwdrivers, needlenose pliers, pocket level, mirror alignment tool

APPENDIX B

ATR MIRROR ALIGNMENT

This appendix provides procedures to follow should the attenuated total reflectance (ATR) mirror need realignment. This material is reproduced from the Spectra-Tech CONTACT SAMPLER™ operator's manual.

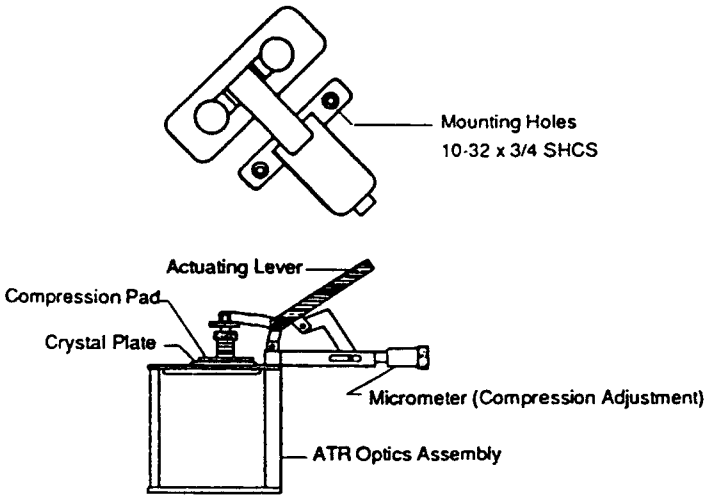
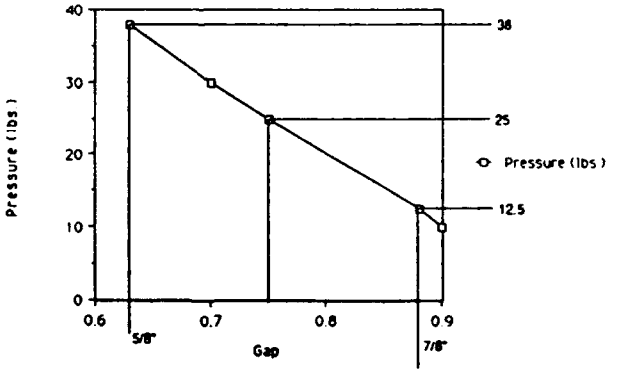


Figure 5: Mounting the Gripper on the Contact Sampler

Figure 6: Pressure vs. Gripper compressed spring height



How to align the Contact Sampler:

1. Verify that a sampling plate is properly installed.

CAUTION: When checking energy throughput through the crystal, the Gripper pressure pad *should not* be in position.
CAUTION: Do not look directly down at the top of an installed Contact Sampler if no sample plate is in position. Laser light is directed up from the transfer mirror through the light pipe and out the top of the accessory. Severe eye damage can result from prolonged exposure to the laser light.

2. Record the energy throughput of the accessory by monitoring either the throughput energy number or the height of the centerburst of the interferogram.
3. Compare this value with the value for the open beam energy throughput, recorded prior to installing the Contact Sampler.
4. If the accessory throughput is 20 - 30% of the open beam throughput (with a new ZnSe crystal), then no further adjustments are required. At this level, the accessory will provide sufficient energy to permit good signal-to-noise ratios using a DTGS detector and moderately short acquisition times (less than one minute.

> NOTE: With a Ge crystal, the energy throughput will be lower. For example, 45° Ge crystals can show throughput as low as 8-10%.

5. If the energy throughput is below 15%, begin to gently rotate the transfer mirrors using the aluminum alignment tool provided. (See Figure 8)

   When making these adjustments, make sure the spectrometer beam (laser or white light) is hitting the first mirror squarely in the middle. Note that on some spectrometers, the laser beam is not located in the center of the IR beam. You should make fine adjustments on each mirror until the energy throughput is maximized. The mirror adjustments should be made in the order listed below. Refer to Figure 9 for the correct numbering sequence for the transfer mirrors.

   - Peak energy for mirror 6
   - Peak energy for mirrors 2 and 3
   - Peak energy for mirrors 4 and 5
   - Peak energy for mirrors 1 and 6

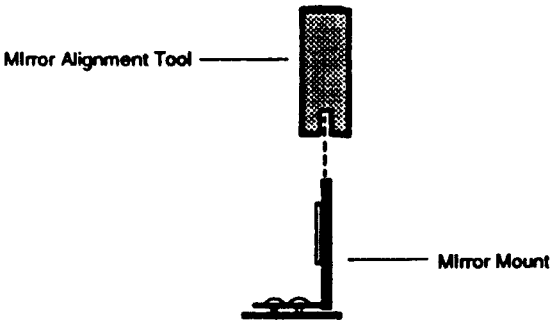


Figure 8: Adjustment of transfer mirrors

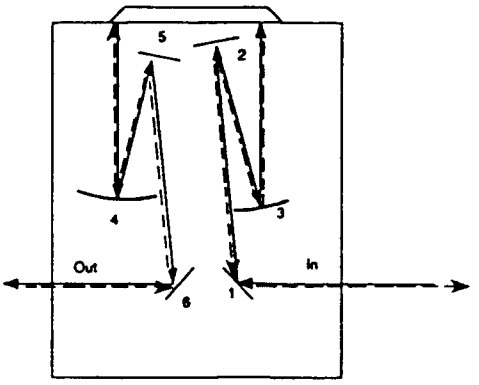


Figure 9: Out-of-Compartment Contact Sampler optical path

6. Following Step 5, if your energy throughput is between 15 and 25%, no further adjustments are required. If your energy throughput is still below that level, however, then proceed with Step 7.
7. Adjust the tilt on the transfer mirror assemblies. While monitoring either the *throughput energy* number or the height of the centerburst of the interferogram, press down gently on the front side of the first transfer mirror bracket. Then, repeat this step while pulling up gently on the rear side of the mirror bracket. (See Figure 10)

   - ❑ If energy increases by applying pressure to the front of the mirror bracket, loosen the front screw and tighten the back screw.
   - ❑ If energy increases by applying pressure to the back of the mirror bracket, loosen the back screw and tighten the front screw.

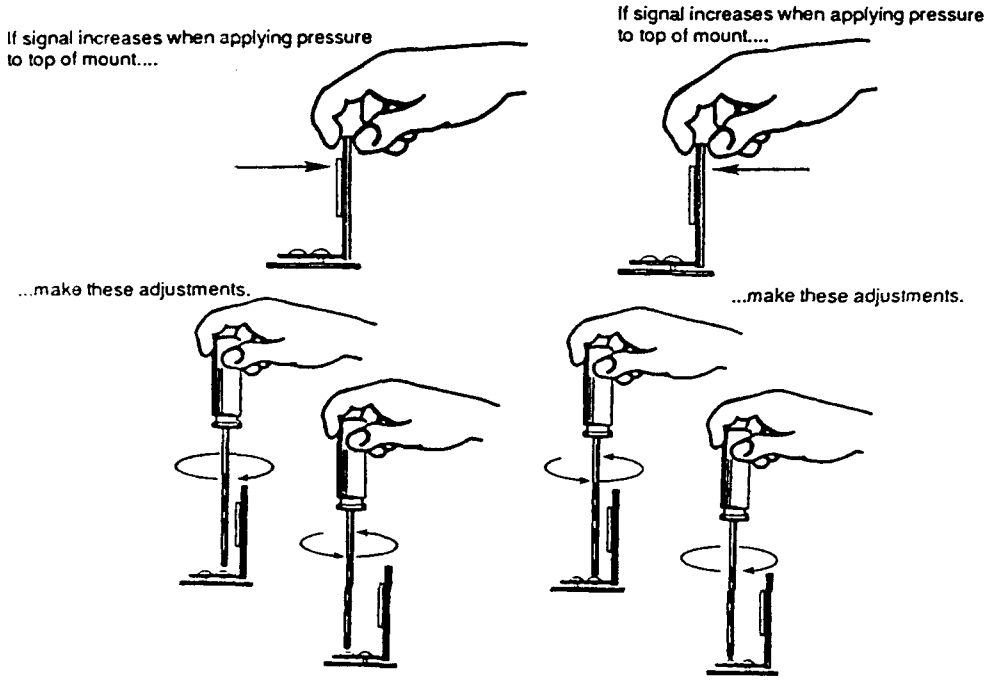


Figure 10: Adjustment of mirror tilt

8. Repeat Step 7 for the other transfer mirror assembly. *Do not attempt to make this adjustment on just one mirror.*
7. Repeat the mirror rotation adjustment (Step 6) to maximize energy throughput. If properly adjusted, throughput should be between 25-30% of the open beam energy reading.

How to install the out-of-compartment purge cover:

The out-of-compartment Contact Samplers are shipped with eiher a Nalgene apron or with a cover plate which can be used to cover the sample compartment opening to maintain purge. The sheet provided is oversize to accommodate whatever configuration you may have, but can be cut to fit neatly on your spectrometer.

1. Unroll the Nalgene sheet and locate the cutout for the spectrometer and the mounting pins. The sheet is marked to identify which end faces front. The marked side goes face down.
2. Place the apron on a flat surface and slip the Contact Sampler module through the cutout. (See Figure 11)

APPENDIX C

OPERATION WITH MATTSON SOFTWARE

ACQUIRING SPECTRA

If you wish to acquire, store, and display spectra independent of the macro program, the Mattson FIRST™ software can be used. This is a general purpose software package for acquiring and saving spectra. It allows you to display and compare several spectra simultaneously and to perform various spectral manipulations. This manual will guide you through only the most basic functions on acquiring, saving, and displaying spectra.

Once the path command in the autoexec.bat file has been modified to include \FIRST\BIN, the Mattson software can be accessed from any directory. If you are planning to store spectra, it is generally best to create a separate directory where spectra will be stored. Move to that directory and then access the FIRST™ software. The FIRST™ software can be access by typing either **acq** for acquire, or **first**. Typing **acq** gets you directly into the data acquisition part of the program, whereas typing first gets you directly into the data analysis part of the program. Once you are in either part of the software, it is very easy to go back and forth between the two parts.

Type **acq** and get into the acquisition program. Selections are made by moving the cross hair or arrow with the mouse, pointing at the desired selection, and then clicking the left-hand mouse button. Either the specific task is executed or another menu is pulled down. First select **Method**. Then pick **Choose an existing Method**. Next, select **method-a**. This Method has been set up at Naval Surface Warfare Center (NAVSWC) and contains the information about the proper resolution, scan velocities, etc., for acquiring the spectra. Next, select the **Scan** window. Then select **Collection Menu**. Then **Initialize** must be selected. After the Initialization Complete message appears, press any key to restore the active screen. If there were difficulties in performing the initialization and you get an Initialization Failed message, hit any key to restore the screen. Often, on the second attempt, the initialization will work; but, it is usually best just to turn the spectrometer off for a few seconds, turn it back on, wait for the self diagnosis, and then try again. Anytime that you leave the first software to return to DOS, **upon returning to the acquisition program, you must re-select the method-a method and re-initialize the spectrometer.**

After initializing the spectrometer, a background spectrum must be taken. To prepare for a background scan with the ATR device, make sure that the crystal is in place and clean. With the pressure plate suspended above the crystal, but not contacting it, select **Background**. After the background scan has been completed, a sample scan can be performed. Prepare samples and load them according to the sections in the **OPERATION** portion of the manual pertaining to **Sample Preparation** and **ATR Sample Loading.**

After loading the sample for scanning, select the **Sample** option from the Collection Menu. The program will ask if you want to overwrite the **tmp.abs** file. By default the stored data from a scan will be written to a file named **tmp.abs**. If you do not wish to save the contents of this file, or if it has already saved under another name, then select **yes**. If you had forgotten to copy the file, select no and copy it now according to the procedure in the **Saving the Spectrum** section.

DISPLAY AND SCALE SPECTRUM

After the scan is complete, the interferograms will be processed and the resulting spectrum will be displayed. Press the left mouse button once to activate the screen. A screen filling pair of cross hairs will appear. If you wish to enlarge any portion of the spectrum, move the cross hairs to one corner of the region and press the mouse button. Then move to the opposite corner and click again; the selected region will now fill the screen. If you wish to restore the original display, select the **Display** window and then select **Reset Display**.

SAVING THE SPECTRUM

If you wish to copy the spectrum into a permanent file, select **Method**, then select **Save Results**. The current name of the file (**tmp**) is given including the complete pathname. Generally, you will want to backspace over **tmp** and write in the desired name. It is not necessary to include the ***abs*** extension. If you wish to exit the program to return to DOS, select **System** then **Quit**. If you wish to call up the FIRST™ software for further data analysis, select **System** then **First**.

ANALYZING SPECTRA

If one wishes to analyze spectra or compare spectra to one another using the Mattson FIRST™ software, type **first** at the DOS prompt or select **System**, then **First** from the acquisition menu. The FIRST™ software can be used to perform many analysis functions, spectral manipulations, and comparisons. No discussion will be given for most of these functions, but the software is quite self explanatory and the use of these functions can be self taught. Discussion will include how to call up spectra and how to display and compare them to one another. If you have forgotten to save the **tmp** file to a permanent file, or if you made changes to a spectrum and want to save it under a new name, you can do it in the FIRST™ software also. To save files, select **Data**, then **Save Results**.

Selecting the Sample

One or several spectra can be viewed at a time. One spectrum is always designated as the sample and the others are designated as references. Choose a sample by selecting **Data**, then **Sample**, then picking the desired file from the list. The spectrum will immediately be displayed in green.

Selecting References

Choose references by selecting **Data**, then **References**, then **Add**. Choose between 1 and 16 files from the list by clicking on each one in turn. Move the arrow outside of the orange list box and click. These files will now be displayed along with the sample spectrum. Additional spectra can be chosen as references at any time, up to a total of 16. References can be removed from the list by selecting **Data**, then **References**, then **Remove**. Select the file to be removed. The spectrum will not be immediately removed from the display. To do so, select **Undo**, then **Reset Display**.

The spectra can be displayed in one of four different formats: sample, stack, superimpose, or overlay. The sample format displays just the sample. Stack divides the screen into equal divisions and displays autoscaled spectra of sample and references within these divisions. Superimpose autoscales all of the spectra to the full screen height. Overlay autoscales the sample spectrum, then overlays the reference spectra on that scale. To choose one of these display formats, select **Display**, then **Format**, then **Style**. Keep clicking on the **Style** selection until your choice appears to the right of **Style**. Click the mouse outside of the orange box, and the spectra in the desired format will be displayed. To expand any portion of the spectrum, move the curser into the plotting area to produce the large cross hairs. For the **Sample** and **Overlay**, modes click on the opposite corners of the box to be expanded and that region will fill the entire screen. For the **Stack** and **Superimpose** modes, you only need to select X-axis endpoints and that region will be expanded. To return to previous expansions, select **Undo**, then **Undo Last Expand** to retrieve the previous display or, **Reset Display** to display the original expansion.

Plotting Spectra

If you wish to plot spectra, you will need to attach a Hewlett-Packard (HP) or compatible plotter (not supplied) with an RS-232 interface to the plotter connection at the rear of the spectrometer. An RS-232 cable is provided for this function. After a plotter is installed, create the display that you desire, then select **Plot** from the menu bar at the bottom of the screen. If one wishes to make annotations to the plot, select **Annotate**. Once the desired display is on the screen, select **Plot** and the plot will be made on the HP plotter.

Exiting the Software

If you wish to exit the FIRST™ software, select **System**, then **Quit**. If you wish to load the **Acquisition** portion of the software, select **System**, then **Data Acquisition**. You can toggle back and forth between the FIRST™ software and the **Data Acquisition** software without having to repeatedly choose the acquisition method or re-initializing the spectrometer.

APPENDIX D

GENERATING A CALIBRATION CURVE

A calibration curve is a plot of the ratio of two Fourier transform infrared (FTIR) peak heights versus the isocyanate (NCO) index. The specific peaks used were found to correlate with the NCO index. Since the calibration curves vary from lot to lot, a new calibration curve is required for each lot of material. The overall procedure is to prepare samples with a range of carefully controlled NCO index values using a batch process and to measure the peak height ratio of these samples. Once the calibration curve has been generated, the NCO index of any material from that lot can be determined from a measurement of peak height ratio.

The materials to be analyzed are made from a mixture of two parts. When the stoichiometric amount of Part A is mixed with Part B so that every NCO group reacts, the mixture is said to have an NCO index of 100, meaning 100 percent of the required amount of isocyanate. Similarly, an NCO index of 95 means there is slightly less than the stoichiometric amount of isocyanate, and an NCO index of 105 means that there is a slight excess of isocyanate. To establish the calibration curve, the following NCO index samples are prepared: 85, 95, 100, 105, 110, 115, and 125.

To obtain reproducible and reliable results, the samples must be properly molded. Machined samples do not give acceptable data. Recall that the ATR technique only probes the surface layer of the sample and, therefore, the sample surface finish is crucial. It is also important that the sample thickness be proper, because samples that are too thick are not only difficult to fit into the attenuated total reflectance (ATR) fixture but often lead to breaking the crystal. Samples that are too thin require readjustment of the holder which is time consuming and can lead to nonreproducible results.

Having prepared samples of the desired NCO index values, the FTIR spectra and peak height ratios are obtained as described in the body of this manual. For spectra collected with the **ratio-a** program, ratios are provided automatically. For spectra collected using the FIRST™ software, the **ratio-b** program calculates the ratios. When values from each of the standard NCO samples have been obtained, plot the data on a graph labeled with the lot identification. Use this plot to determine NCO index for all samples made from this lot.

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13. ABSTRACT *(Maximum 200 words)*

This manual contains detailed instructions for the operation of a Fourier transform infrared (FTIR) spectrometer. The instructions cover the installation of the spectrometer and its associated computer after shipment, the operation of the instrument, and repackaging for shipment. Some hints on troubleshooting are also provided.

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